

# Superconductivity in $MgB_2$ doped with $Ti$ and $C$

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## Abstract

Measurements of the superconducting upper critical field,  $H_{c2}$ , and critical current density,  $J_c$ , have been carried out for  $MgB_2$  doped with  $Ti$  and/or  $C$  in order to explore the problems encountered if these dopants are used to enhance the superconducting performance. Carbon replaces boron on the  $MgB_2$  lattice and apparently shortens the electronic mean free path of  $MgB_2$  and raising  $H_{c2}$ . Titanium forms precipitates of either  $TiB$  or  $TiB_2$  that enhance the flux pinning and raise  $J_c$ . Most of these precipitates are intra-granular in the  $MgB_2$  phase. For samples containing both  $C$  and  $Ti$  doping, the  $C$  appears to still replace  $B$  in the  $MgB_2$  lattice and the  $Ti$  precipitates out as a boride. If approximately 0.5% $Ti$  and approximately 2% $C$  are co-deposited with B to form doped boron fibers and these fibers are in turn reacted in  $Mg$  vapor to form doped  $MgB_2$ , the resulting superconductor has  $\mu_0 H_{c2}(T=0) \sim 25\ T$  and  $J_c \sim 10,000\ A/cm^2$  at 5 K and 2.2 T.

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## I. INTRODUCTION

The performance of  $MgB_2$  superconducting materials[1] can be greatly enhanced by the addition of small amounts of carbon that will raise the upper critical magnetic field,  $H_{c2}$ , [2] and the critical current density,  $J_c$ . [3] Point defects like carbon that substitute for boron in the host lattice have been shown to raise  $\mu_o H_{c2}(T = 0)$  from 16 T in pure  $MgB_2$  to 32 T for a carbon level of  $\sim 4\%$ . [4] At higher carbon contents,  $\mu_o H_{c2}$  rises more slowly and eventually drops to about 25 T at  $\sim 10\%$  carbon. [5, 6] Several different precipitate phases have been used to form small pinning sites in the  $MgB_2$  lattice. For example,  $SiC$ , [7]  $YB_4$ , [8],  $TiB_2$  [9] and  $MgSi_2$  [10] have been added and show a rise of  $J_c$  into the range of  $10^6 A/cm^2$  at 10 K and self field with about 5% precipitate additions. In many of these experiments, either a powder-in-tube (*PIT*) process or a pressed and sintered pellet method have been used to form the precipitates for the synthesis.

In a different approach to sample preparation, a chemical vapor deposition, *CVD*, method [11] can be used to co-deposit  $B$  together with a doping element to form long lengths of carbon doped boron fiber. [4] Subsequent heat treatment in  $Mg$  vapor transforms the doped boron into doped  $MgB_2$ . [4] Both the *CVD* and powder methods have advantages. The advantage of powder methods is that the diffusion lengths are comparable to the powder size giving relatively low reaction temperatures and short reaction times. The advantage of co-depositing the impurity with the  $B$  in a *CVD* process is that the impurity is mixed with the  $B$  on an atomic scale. Both  $Ti$  [3] and  $C$  [4] separately have been successfully doped into  $MgB_2$  using these *CVD* methods.

The purpose of the work reported here is to study the combined  $C$  and  $Ti$  doping of  $MgB_2$  to determine whether samples can be prepared with the combined benefits of both dopants, raising  $\mu_o H_{c2}$  to the range of 30 T and raising  $J_c$  values to the range of  $10^4 A/cm^2$  at 20 K and 5 T. Two specific questions need to be addressed. Does the addition of  $Ti$  to the  $C$ -doped  $MgB_2$  samples reported earlier, [4] reduce the amount of  $C$  in the  $MgB_2$  lattice, possibly by forming  $TiC$  precipitates? In the presence of  $C$ , can the  $Ti$  precipitate size be maintained at  $\sim 5 nm$  for high  $J_c$  performance?

## II. EXPERIMENTAL

Long lengths of doped boron fiber are prepared in a *CVD* apparatus similar to that used for commercial boron filament production.[11] All of the materials reported here were deposited on a *W* fiber with an initial diameter of about  $15\mu m$ . The *W* enters the reaction chamber moving at a few *cm/s* through a *Hg* seal into a long glass tube containing flowing  $H_2$ ,  $BCl_3$ ,  $CH_4$ , and  $TiCl_4$ . For *Ti* doping,  $H_2$  is bubbled through liquid  $TiCl_4$  to provide  $TiCl_4$  molecules in the gas stream. Typically the  $BCl_3$  flow rate was  $3\text{ l/min}$  with the  $CH_4$  and  $TiCl_4$  flow rates adjusted for the desired doping levels. The diameter of the exiting doped *B* fibers were in the  $75\mu m$  to  $100\mu m$  range. The doped *B* fibers were cut to lengths of  $\sim 3\text{ cm}$  and placed in a *Ta* tube with a *Mg* to *B* ratio of 1 : 1. The *Ta* was welded shut, sealed in a quartz tube, and heat treated in a box furnace for appropriate periods. Upon removal from the furnace, ampoules were quenched in water. Resistivity measurements are made by a four contact method using silver epoxy to make the electrical contacts. A Quantum Design *PPMS* system was used to make resistivity vs temperature,  $\rho$  vs.  $T$ , measurements up to  $14\text{ T}$ . At higher fields,  $\rho$  vs.  $H$  measurements are made in a  $32.5\text{ T}$  copper coil magnet at the National High Magnetic Field Laboratory at Florida State University. Magnetization measurements were made in a Quantum Design SQUID magnetometer with a magnetic field range of  $5.5\text{ T}$  and  $J_c$  was determined from the magnetization hysteresis using the Bean Model for cylindrical shell samples.[3] A Philips CM30 transmission electron microscope (*TEM*) was employed for the microstructure characterization. Samples for the *TEM* were made using a crush-flow technique.

## III. *Ti*-DOPING ONLY

In an earlier publication,[12] we investigated a series of *Ti*-doped  $MgB_2$  samples that were deposited on a commercial carbon coated *SiC* fiber substrate with a diameter of approximately  $80\mu m$  instead of the  $15\mu m$  diameter *W* substrate used here. The doped- $MgB_2$  layers ranged from about  $4\mu m$  to  $10\mu m$  thick. Flux pinning was excellent and gave  $J_c$  over  $10,000\text{ A/cm}^2$  at  $25\text{ K}$  and  $1.3\text{ T}$  for a sample with an average doping of about  $9\%Ti$ . [3] Unfortunately, there were problems with this approach to sample preparation. First, the diffusion of *Mg* into the *B* caused swelling and the  $MgB_2$  pulled away from the substrate.

Second, the presence of a carbon coated substrate made the amount of  $C$  in the specimen uncertain. In addition, these samples had a rather inhomogeneous  $Ti$  distribution. There was a high  $Ti$  level near the  $SiC$  core and near the outer surface of the fiber.[3] Transmission electron microscope ( $TEM$ ) observations for one of these samples that had been reacted for 2 h at  $950^\circ C$  are shown in Fig. 1a. Results reveal a random distribution of precipitates scattered through the grain, ranging in size from 1 nm to 20 nm with a spacing about 5 times as large. Energy dispersive spectroscopy for a large portion of the grain in Fig. 1a, revealed that the  $Ti/Mg$  ratio was  $\sim 5\%$ . Selected area diffraction taken along the c-axis showed the prominent hexagonal pattern of  $MgB_2$  with additional powder pattern rings arising from the titanium boride precipitates. The beam was then tipped off axis to show the rings more prominently. Indexing the rings revealed that the precipitates were  $TiB$  rather than  $TiB_2$ . These results differ from those found by Zhao and coworkers.[13] They found that samples prepared by mixing powders of  $Mg$ ,  $Ti$ , and  $B$  and reacting gave  $TiB_2$  precipitates on the  $MgB_2$  grain boundaries. This illustrates that different sample preparation methods can yield different types and locations of the precipitates. Both the precipitation of  $TiB$  throughout the grains[12] and precipitation of  $TiB_2$  on the grain boundaries[13] seem to give enhanced flux pinning.

Samples with 15  $\mu m$  diameter tungsten cores were prepared using three different  $TiCl_4$  flow rates in the  $CVD$  reactor, 0.42 cc/min, 1.26 cc/min and 2.9 cc/min. After the  $CVD$  deposition, the fibers were reacted in  $Mg$  vapor to form  $Ti$ -doped  $MgB_2$ . Energy dispersive spectra (EDS) in a scanning electron microscope (SEM) was used to probe the uniformity of  $Ti$  distribution across the superconducting fiber and to measure the  $Ti$  to  $Mg$  ratio,  $[Ti]/[Ti + Mg]$ . Line scans in various regions of the sample show that the  $Ti$  level was uniform to about 10% of the average value. Multiple point scans and area scans in the EDS give average values of the  $[Ti]/[Ti + Mg]$  ratio, shown in Fig. 2 indicating that the percent  $Ti$  is roughly linearly related to the flow rate in the reaction chamber. Point scans, line scans, and large area raster scans were used for analysis. The large area raster scan shown by the open squares on Fig. 2 comprises the most data and are probably the most accurate measure of the  $Ti$  content. These three flow rates of 0.42 cc/min, 1.26 cc/min, and 2.9 cc/min, give 0.3%, 0.5% and 1.6% respectively for the  $[Ti]/[Ti + Mg]$  ratio.

Because these  $Ti$  doped samples were deposited on a 15  $\mu m$  diameter  $W$  wire and had a 76  $\mu m$  outer diameter, these fibers require much longer times or higher temperatures to

fully form the  $MgB_2$  phase than the 4 to 10  $\mu m$   $B$  layers of the earlier work.[3] Therefore, temperatures of 1000 to 1200°C were often used. A sample with 0.5% $Ti$  reacted for 72  $h$  at 1000°C were found to be 95% reacted using polarized light in an optical microscope. A  $TEM$  micrograph of this sample, Fig. 1b, shows precipitates that are much larger than in Fig. 1a. In this figure, the sweeping shaded areas arise from the underlying holey carbon support. In this micrograph, the beam has been tilted to emphasize areas of dense dislocations as shown, for example, in the top center of the micrograph. Many of the precipitates were 50 to 200  $nm$  in diameter. Selected area diffraction indicated the precipitates were  $TiB_2$  and had c-axes coaxial with the  $MgB_2$  grains in which they were imbedded. For a sample with 0.5% $Ti$  plus 2.1% $C$ , the sample was fully reacted after 48  $h$  at 1200°C and shows the 20 to 100  $nm$  diameter precipitates of Fig. 1c. Again the large precipitates here are  $TiB_2$  with the c-axis parallel to the c-axis of the  $MgB_2$  grain in which it is imbedded.

Another series of samples having 0.3%, 0.5% and 1.6%  $Ti$  were all reacted at 1100°C for 48  $h$  and studied in the  $TEM$ . For 0.3% $Ti$ , the precipitate size is less than 50  $nm$  and widely spaced. For 0.5% $Ti$ , the precipitate size ranges from 20 to 80  $nm$ . For 1.6% $Ti$ , the precipitate size ranges from 20 to 100  $nm$  and the density of precipitates is correspondingly higher. All of these samples show  $J_c$  values higher than pure  $B$ , but lower than shown by the sample of Fig. 1a.

Magnetization data, shown in Fig. 3, indicate that the suppression of  $T_c$  with  $Ti$  doping depends on the temperature at which the  $MgB_2$  forms. Samples reacted at 1000°C for 72  $h$  have magnetization curves similar to pure  $MgB_2$  except that they are shifted to lower temperature by about 1  $K$ . The 0.3% $Ti$  and 1.6% $Ti$  are nearly identical whereas the 0.5% $Ti$  sample is a bit lower. The depression is not monotonic in  $Ti$  content, and the results on Fig. 3 probably represent true scatter in the data. The cause of the suppression in  $T_c$  is not understood. If the  $Ti$  all precipitates as  $TiB$  or  $TiB_2$  and no  $Ti$  is incorporated in the  $MgB_2$  lattice, then it might be expected that the suppression of  $T_c$  might be rather small and arise from lattice strains induced by the precipitates or other defects in the  $MgB_2$ . It is also possible that some  $Ti$  atoms replace  $Mg$  in the  $MgB_2$  lattice and reduce the superconducting interaction in that way. Further study is going to be needed to determine which variables contribute to this suppression in  $T_c$ .

At 1100°C for 48  $h$ , the suppression of  $T_c$  behavior in Fig. 3b is similar to Fig. 3a except that the downward shift of  $T_c$  is somewhat larger. At 1200°C for 24  $h$ , shown in Fig. 3c,

the suppression of  $T_c$  is very large and increases monotonically with  $Ti$  content. Because the  $4\pi M$  vs.  $T$  curves are not always monotonic in the  $Ti$  content, a new series of samples were made at  $1100^\circ C$  to check reproducibility. The data for the two sets of samples with the same  $Ti$  content differed by as much as  $0.5\text{ K}$ . With these large  $TiB_2$  precipitates, there are some as yet uncontrolled parameters.

An X-ray study of the shift in the  $MgB_2$  a-axis lattice constant was undertaken to look for a connection between the lattice constant and the amount of impurity. For the case of carbon,[4] the a-axis lattice constant contracts linearly with increasing  $C$ , as shown by Fig. 4, while the c-axis remains essentially unchanged as reported by several authors.[4] For the case of  $Ti$ , the picture is more complicated. As shown in Fig. 4, the a-axis contracts with increasing  $Ti$ , but there is considerable scatter in the data and the amount of change in the a-axis may depend on the temperature at which the  $MgB_2$  is formed. An additional sample with  $4.7\text{ cc/min}$  flow rate of  $TiCl_4$  ( $2.4\%Ti$ ) and reacted at  $1200^\circ C$  for  $12\text{ h}$  is shown by the open square of Fig. 4. The apparent flat region of the a-axis lattice parameter between  $0.5\%$  and  $2.4\% Ti$  as shown by the open squares would appear to indicate that a solubility limit has been reached for  $Ti$  in  $MgB_2$  at about  $0.5\% Ti$ .

For the case of  $Ti$  additions only, samples with an  $1100^\circ C$  for  $48\text{ h}$  reaction temperature are used to illustrate the changes in superconducting properties. The values of  $H_{c2}$  measured up to  $9\text{ T}$  for the three different  $Ti$  concentrations are slightly lower than for pure  $MgB_2$  as shown on Fig. 5. As was found for values of  $4\pi M$  vs.  $T$  curves in Fig. 3b, the  $0.5\% Ti$  sample has the largest suppression of  $T_c$  and the largest suppression of  $H_{c2}$ .

As shown by Fig. 6, the  $J_c$  values at  $20\text{ K}$  for these three  $Ti$  concentrations are enhanced by about a factor of 10 at fields, up to about  $1.5\text{ T}$ . There is very little difference in  $J_c$  as the  $Ti$  level is raised from  $0.3\%Ti$  to about  $1.6\%Ti$ . Values of  $J_c$  drop through  $1\text{ kA/cm}^2$  at about  $1.5\text{ T}$  even though  $\mu_o H_{c2}$  is above  $6\text{ T}$  at  $20\text{ K}$  for these three samples as shown in Fig. 5.

#### IV. COMBINED $Ti$ AND $C$ DOPING

All the samples reported here for the combined doping were reacted at  $1200^\circ C$  to ensure full conversion to the  $MgB_2$  phase. The  $C$  content is determined from the  $CH_4$  flow rate in the  $CVD$  chamber and previous results.[4] For a sample with  $0.5\%Ti$  plus  $2.1\%C$  shown in

Fig. 1c, the precipitates are 20 to 100 *nm* in diameter and selected area diffraction shows the precipitates to be  $TiB_2$  with the c-axis parallel to the c-axis of the  $MgB_2$  grain in which they are imbedded. In this micrograph, the beam is tilted so that the dislocations are not so apparent, but they are there. These precipitates of Fig. 1c are similar to those in Fig. 1b, but in contrast to the  $TiB$  precipitates of Fig. 1a.

An X-ray analysis of the combined  $Ti$  and  $C$  doping is illustrated in Fig. 7. The c-axis lattice parameter from the (002) peak essentially does not change with  $Ti$  and  $C$  addition. The a-axis lattice parameter contracts in a regular way as shown by the (110) peak of Fig. 7. The 0.5%  $Ti$ -only peak moves to higher angle than the pure  $MgB_2$  peak by about 0.11 degree as shown by the heavy dotted line. The 2.1%  $C$ -only peak shifts out from the pure  $MgB_2$  peak by about 0.22 degree as shown by the light dotted line. And, the 0.5%  $Ti$ +2.1%  $C$  peak shifts out from the pure  $MgB_2$  peak by about 0.29 degree as shown by the heavy solid line. Roughly speaking, the decrease in a-axis lattice parameter is additive for  $Ti$  and  $C$  doping at this level.

As was reported earlier,[4] the  $4\pi M$  *vs.*  $T$  curves and the resulting  $T_c$  values are depressed monotonically with increasing  $C$  content as shown by the open squares and solid circles of Fig. 8. If an additional 0.5%  $Ti$  is added to each of these  $C$  concentrations, the combined suppression of  $T_c$  is roughly additive. As shown in Fig. 8, the addition of 2.1%  $C$  to  $MgB_2$  suppressed  $T_c$  by about 2  $K$  and the addition of 2.1%  $C$  plus 0.5% $Ti$  suppressed  $T_c$  by about 6  $K$ .

Values of  $\mu_o H_{c2}$  in samples with the combined doping shown in Fig. 9 by the open circles are very similar to values for  $C$  doping only reported previously[4] as shown by the solid symbols. The solid triangles were taken with  $R$  *vs.*  $T$  measurements and the solid circles were taken at the National High Magnetic Field Laboratory as  $R$  *vs.*  $H$  measurements. For the combined  $Ti$  and  $C$  doped samples there was considerable rounding at the high field end of the  $R$  *vs.*  $H$  transitions. The two open circles represent two different definitions of  $\mu_o H_{c2}$ , the lower being a linear extrapolation of the long linear region of  $R$  *vs.*  $H$  up to the normal state, and the upper open circle being the field where the resistivity reaches the normal state value within the noise. The addition of a few percent  $C$  to the  $Ti$  doped samples substantially raises  $\mu_o H_{c2}$  (open circles) to values comparable to values for carbon only (solid symbols).

Values of  $J_c$  for a series of samples reacted at 1200°C are shown in Fig. 10. For the

combined 2.1%  $C$  + 0.5%  $Ti$  sample at 5  $K$ , the  $J_c$  curve crosses  $1\text{ kA/cm}^2$  at about 3.2  $T$  shown by the solid circles. The 1.1%  $C$  + 0.5%  $Ti$  sample at 5  $K$  crosses  $1\text{ kA/cm}^2$  at 2.6  $T$ , as shown by the open circles. The 0.5% $Ti$  only sample has the highest low field  $J_c$  values and crosses  $1\text{ kA/cm}^2$  at 2.3  $T$ . A pure  $MgB_2$  sample at measured at 5  $K$  is shown by the solid squares. It is not shown here, but the the combined 2.1%  $C$  + 0.5%  $Ti$  sample at 20  $K$ , the  $J_c$  curve crosses  $1\text{ kA/cm}^2$  at about 1.5  $T$ .

To summarize, the addition of  $C$  to raise  $H_{c2}$  and the addition of  $Ti$  to form precipitate pinning centers are roughly independent of one another for the samples reported here. Carbon doped  $MgB_2$  shows a rapid rise in  $\mu_o H_{c2}(T = 0)$  from 16  $T$  for pure  $MgB_2$  to 25  $T$  for  $\sim 2.1\%$  added carbon. With both 2.1%  $C$  and 0.5%  $Ti$  the sample retains a  $\mu_o H_{c2}(T = 0)$  of  $\sim 25\text{ T}$  and no evidence for the formation of  $TiC$  was seen. The addition of  $Ti$  enhances  $J_c$  substantially in the magnetic field range of 2.5 to 3.5  $T$ , as shown in Fig. 10.

Much work needs to be done to optimize the the  $C$  and  $Ti$  levels and the processing to raise  $J_c$  at high fields. For low reaction temperatures and short times to form the  $MgB_2$  phase,  $TEM$  data show that the precipitates are intra-granular and randomly oriented particles ranging in size from 1 to 20  $nm$ . For higher reaction temperatures in the range from 1000 to 1200°C, the precipitates are much larger. The precipitates are usually intra-granular  $TiB_2$  particles coplanar with the  $MgB_2$  host that range from 50  $nm$  to 200  $nm$  in size. Coarsening of the  $TiB_2$  precipitates at high temperatures and long times is clearly a problem. In magnetic fields from zero to 1  $T$ , the addition of carbon to  $Ti$ -doped  $MgB_2$  gives relatively little change in  $J_c$ , but in the 3 to 4  $T$  range, carbon additions clearly enhance  $J_c$ . A very practical problem is that the addition of either  $C$  or  $Ti$  slows down the rate at which the  $B$  fibers transform to the  $MgB_2$  phase. For high  $J_c$  values, it is helpful to react at low temperatures to give small  $MgB_2$  grains and to prevent the  $Ti$  precipitate coarsening. Some method is needed to overcome these slow reaction rates, probably the use of fine powders to keep the reaction time short and the reaction temperatures low. It would be desirable to make doped boron powders so that the diffusion lengths can be much smaller and the reaction temperatures lower.



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## FIGURE CAPTIONS

Fig. 1 *TEM* micrograph of  $MgB_2$  with 5%*Ti* reacted  $950^\circ C$ -2 *h*, *C* coated *SiC* substrate.  
b)  $MgB_2$  with 0.5%*Ti* reacted  $1000^\circ C$ -72 *h*, *W* substrate. c)  $MgB_2$  with 0.5%*Ti* + 2.1%*C* reacted  $1200^\circ C$ -48 *h*, *W* substrate.

Fig. 2 Percent *Ti* in  $MgB_2$  sample as a function of the  $TiCl_4$  flow rate in the *CVD* apparatus.

Fig. 3 Magnetization transitions for 3 different *Ti* levels at a)  $1000^\circ C$ , b)  $1100^\circ C$  and  $1200^\circ C$ .

Fig. 4 Comparison of change in a-axis lattice constant for both *Ti* and *C* doping.

Fig. 5  $H_{c2}$  for  $MgB_2$  doped with *Ti* only.

Fig. 6 Enhancement of  $J_c$  with *Ti* additions.

Fig. 7 X-ray data for the (002) and (110) peaks for pure B, 0.5% *Ti*, 2.1%*C*, and 0.5%*Ti* + 2.1%*C*.

Fig. 8 Comparison of *C* + *Ti* doping with *C* only.

Fig. 9  $H_{c2}$  for combined *C* + *Ti* doping (open symbols) compared to *C* only doping (solid symbols).

Fig. 10 Enhancement of  $J_c$  for combined *Ti* and *C* additions. The *Ti* only curve was reacted  $1200^\circ C$  12*h*. The *Ti* plus *C* samples were reacted at  $1200^\circ C$  48*h*.

This figure "Fig1.jpg" is available in "jpg" format from:

<http://arXiv.org/ps/cond-mat/0411531v1>



















